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DEVELOPMENT OF A PROTOTYPE REGENERABLE
CARBON DIOXIDE ABSORBER

BY Michael Onischak

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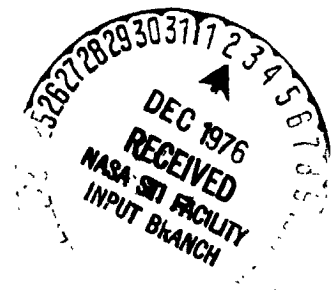
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ENERGY RESEARCH CORPORATION

prepared for

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

NASA Ames Research Center
Contract NAS2-8644



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ABSTRACT

Design information was obtained for a new, regenerable carbon dioxide control system for extravehicular activity life support systems.

Solid potassium carbonate was supported in a thin porous sheet form and fabricated into carbon dioxide absorber units. Carbon dioxide and water in the life support system atmosphere react with the potassium carbonate and form potassium bicarbonate. The bicarbonate easily reverts to the carbonate by heating to 150°C.

The methods of effectively packing the sorbent material into EVA-sized units and the effects of inlet concentrations, flowrate, and temperature upon performance were investigated.

The cycle life of the sorbent upon repeated thermal regenerations was demonstrated through 90 cycles.

1. INTRODUCTION

Future manned space exploration missions are expected to include requirements for astronaut life support equipment capable of repeated use and regeneration for many extra-vehicular activity (EVA) sorties.

The purpose of this program was to design and test a regenerable environmental carbon dioxide removal unit for EVA duty. The method of carbon dioxide removal is new and is based upon a previous study(1,2) that demonstrated its feasibility. The system utilizes the reaction chemistry of solid potassium carbonate forming solid potassium bicarbonate for removal and control of environmental carbon dioxide. Potassium carbonate, a safe, non-toxic chemical, reacts with carbon dioxide and water present in the ventilating gas to form potassium bicarbonate, which by the application of heat reverts back to the carbonate. The regeneration is completed at the low regeneration temperature of 150°C.

It was demonstrated (1) that carbon dioxide concentrations could be maintained below 0.76 mm Hg partial pressure by solid potassium carbonate dispersed and bound into thin (1mm), pliable sheet material. The sheet sorbent form achieved sufficiently fast carbon dioxide reaction rates, good uptake capacity, and was capable of repeated use up to fifty cycles.

To gain understanding of the carbon dioxide sorption and regeneration processes in mission-size units, this study investigated the most suitable method of packing the sorbent material into compact absorber units with gas accessibility to the sorbent.

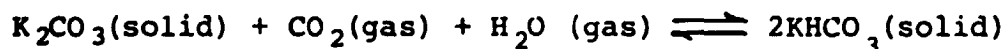
Information about the effects of unit shape, sorbent sheet thickness, gas channel width, and the heat of reaction were obtained for the design of prototype system units. Further, two basic methods of regenerating the sorbent material after sorption of carbon dioxide were explored to identify possible system advantages of one method over the other. The two methods are thermal regeneration by heating to 150°C and regeneration by applied vacuum.

2. BACKGROUND

Although the words sorbent and absorber are used to describe the carbon dioxide removal material and removal unit, respectively, the device is basically a chemical reactor with carbon dioxide bearing gas contacting a solid, consumable

reactant, potassium carbonate. To achieve fast reaction rates and high utilization in the system with flowing gas streams containing rather low concentrations of carbon dioxide, the reactant is dispersed among a high surface area material and bound in place to form a thin porous sheet. The bicarbonate product formed is regenerated in place.

The basic reversible reaction taking place is as follows:



The high porosity and void volume of the sorbent material structure allow practical removal of carbon dioxide repeatedly. Exit carbon dioxide concentrations below 1.0 mm Hg partial pressure are achieved. Partial pressures near zero are thermodynamically possible at room temperature.

In manned system atmospheres, water vapor is present. Water is necessary for the above reaction and an excess of water over carbon dioxide is normally present and advantageous for carbon dioxide removal. This system therefore helps to remove water from the ventilating gases. Furthermore, potassium carbonate forms a hydrate $\text{K}_2\text{CO}_3 \cdot 3/2\text{H}_2\text{O}$ as a side reaction in the system.

Water removed from the system with the carbon dioxide unit benefits the life support system humidity control device, reducing its load and size. The non-regenerable lithium hydroxide canisters used to remove carbon dioxide in previous life support systems added water to the system.

A typical life support system is shown in Figure 1 with the various devices functionally described and in their most probable location. The atmosphere gases are processed through the system with makeup oxygen added before returning to the suit.

3. DESCRIPTION OF ABSORBER TEST STATION

A test facility was constructed to obtain performance data of absorber units under varied conditions representing EVA duty. As shown schematically in Figure 2, carbon dioxide gas was mixed with air to simulate suit vent gases. The concentration of carbon dioxide and water vapor in the air streams were continuously monitored by infrared analyzers.

The test station could provide atmospheric air streams to the absorber units with carbon dioxide concentrations from 1.0 to 40 mm Hg partial pressure at flow rates up to 283 liters per minute STP.

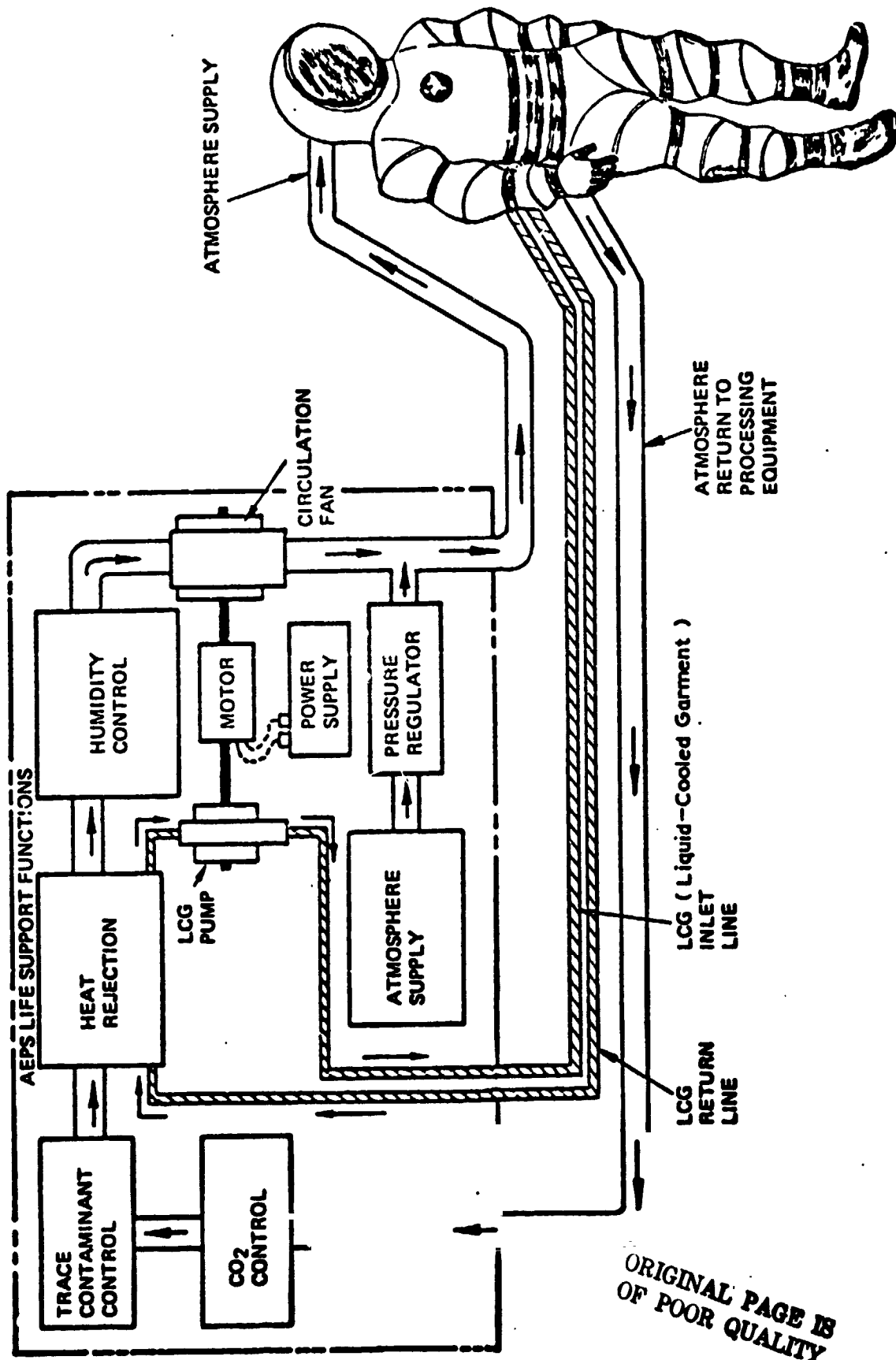


FIGURE 1 LIFE SUPPORT SYSTEM SCHEMATIC (ADVANCED EXTRAVEHICULAR PROTECTIVE SYSTEMS, AEPS) (3)

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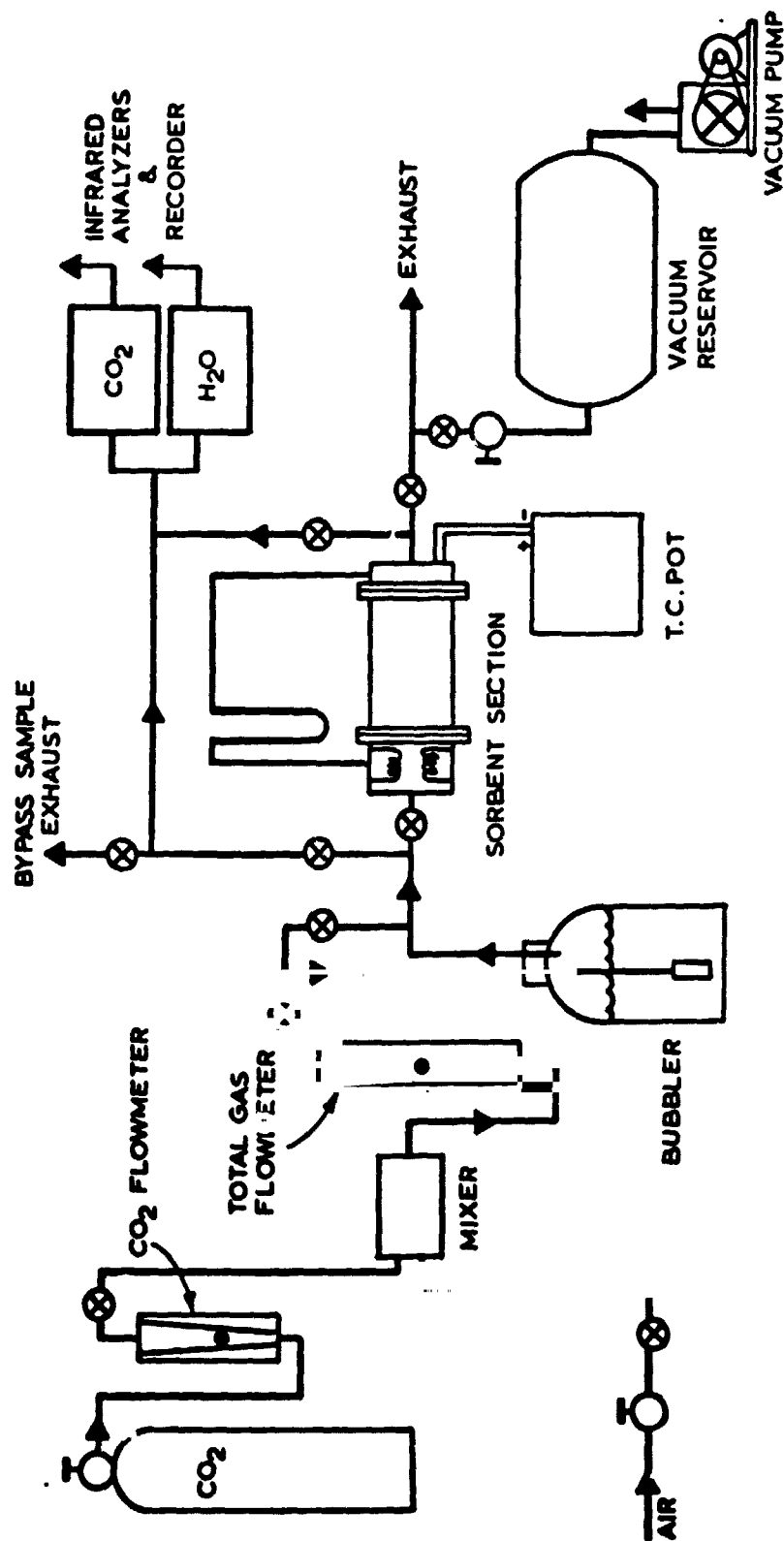


FIGURE 2 ABSORBER TEST FACILITY SCHEMATIC

All testing was done at atmospheric pressure. The sorbent was regenerated thermally at 150°C with electrical heaters surrounding the absorber units. A large volume tank and a high capacity vacuum pump comprised the vacuum reservoir for vacuum regeneration. Fine wire thermocouples were embedded in various zones inside the absorber unit to monitor temperatures. The pressure drop across the absorber was also measured.

The general carbon dioxide test input rate used for absorber testing and evaluation was estimated as 40 liters of carbon dioxide per hour. This was obtained from an estimated metabolic heat load of 202 kcal per hour.

4. SORBENT MATERIAL PREPARATION

The active sorbent material contains reagent grade solid potassium carbonate dispersed among high surface area silica gel and bound in place with a polytetrafluorethylene (PTFE) binder. An optimum sorbent composition was determined in the previous feasibility study consisting of 44 weight percent potassium carbonate, 50 weight percent silica gel, and 6 weight percent PTFE. That optimum sorbent composition removed the maximum amount of carbon dioxide for the least total sorbent weight and was capable of repeated regeneration.

The preparation process of the sorbent material for the absorber test units in this study was scaled up beyond hand prepared laboratory quantity procedures. A machine processing, blending, and calendaring (sheet producing) process was developed. The sorbent material could be prepared in various thicknesses, widths, and lengths. Typically, the sheet dimensions were 1.14 ± 0.076 mm in thickness, steel rule die cut to 14.61 ± 0.076 cm in width, and cut into lengths of approximately 1.0 meter.

A minor sorbent composition change was made resulting from the initial sorbent testing phase. The binder concentration was reduced from six to three weight percent. This change did not alter the carbon dioxide absorption performance but made the material stable upon repeated regenerations. With six percent binder, a residual stress-type behavior was observed which during regeneration heating was relaxed, causing about 10 to 15 percent sorbent shrinkage.

5. ABSORBER UNIT CONSTRUCTION

An axial flow cylindrical design was chosen for the absorber unit because of construction simplicity, simple flow

distribution and radial symmetry. A cylindrical sorbent material core was constructed and inserted in a metal test canister. The sorbent core was made by spirally winding the long, thin sorbent sheets with a corrugated aluminum expanded-metal screen. The peak-to-peak corrugations of the aluminum separator screen formed the axial gas flow channels. A typical absorber test unit is shown in Figure 3. The approximate channel space between sorbent sheets was 0.64mm, and the average sorbent thickness was 1.14mm. Aluminum is lightweight and a good conductor of heat.

Pressure drops through a typical 15 cm channel length varied between 2.0 and 4.4 cm of water pressure for flows up to 113 liters per minute STP.

Various types of expanded metal and screen aluminum separator materials were investigated. They varied in weight, thickness, open area, and corrugation size and pattern. The optimum separator should have the lowest weight and smallest corrugation size that yields the maximum sorbent packing density. Physically, the separator must not damage the sorbent while it holds the core geometry stable.

The thickness of the sorbent material was held constant during all testing. It was increased by 50% over the optimum sorbent found in the previous study because gas in the absorber units contacts the sorbent from both sides.

After fabricating many cores, a stable physical arrangement was found. The aluminum separator material used was 0.203mm thick, 1145 aluminum base metal sheet, pierced and expanded to form a screen-like material with an open area of approximately 62%. The weight is about 0.021 grams per square centimeter.

The units so constructed and tested were stable and capable of repeated regeneration. They are nearly optimum in terms of practical sorbent packing density. However, if a stronger, lighter weight, but rigid separator were found then unit weight could be reduced further. Presently, the 1.3 hour test units contain approximately 730 grams of sorbent and 700 grams of separator, and are 15.2 cm long and 15.2 cm in diameter.

6. ABSORBER PERFORMANCE

Preliminary performance results with respect to sorbent sheet stability, flow distribution, and separator effectiveness were obtained with absorber units of approximately 1.3 hours capacity. The capacity number is used as a reference value

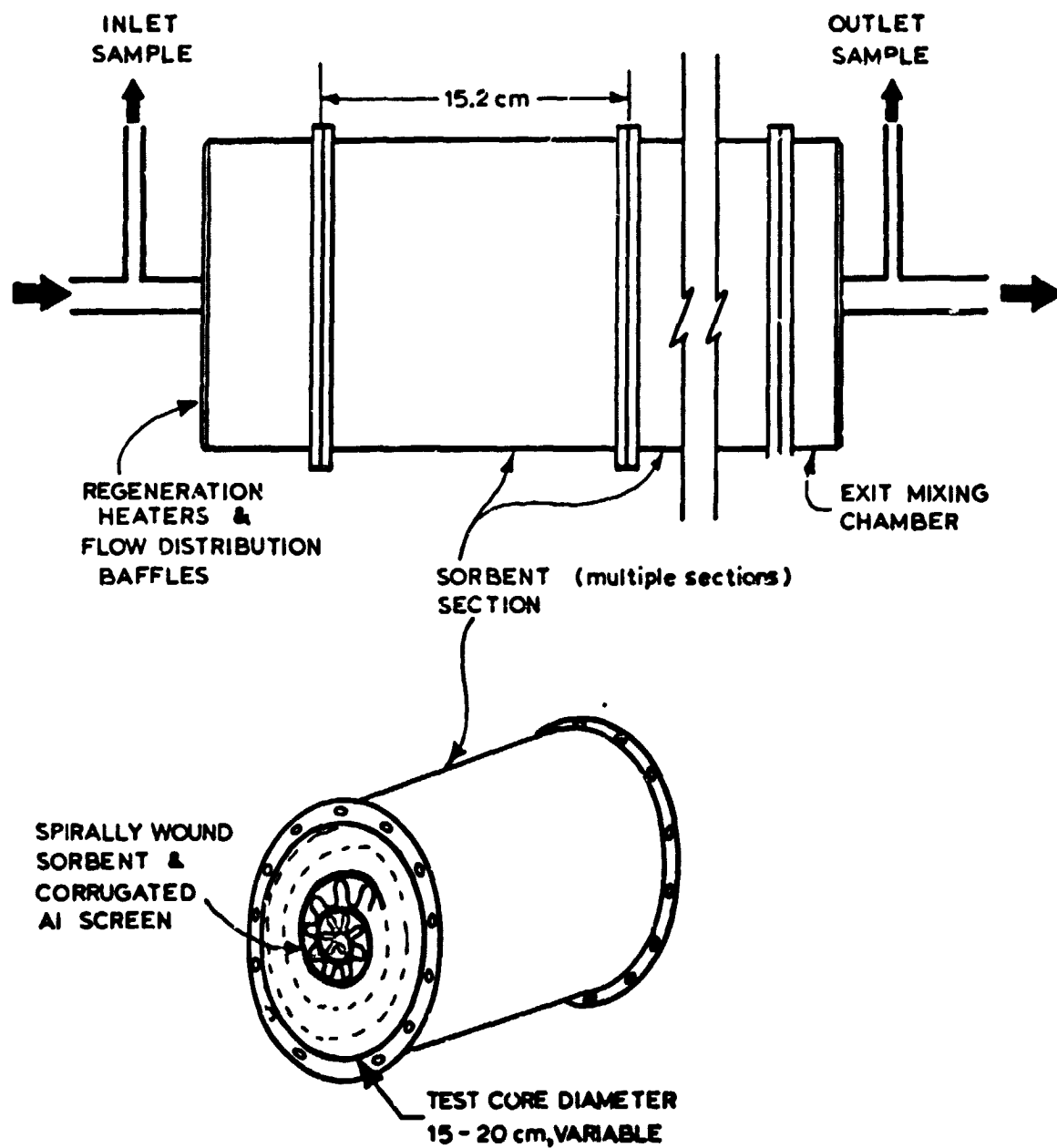


FIGURE 3 ABSORBER TEST UNIT

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and is calculated from the amount of potassium carbonate required to remove 40 liters of carbon dioxide (STP) per hour at 90% conversion of the potassium carbonate.

The size of the unit was conveniently small so that construction time was minimal yet large enough to indicate typical performance.

After the initial investigation for a physically stable core of sorbent and separator, performance testing, including cycle life, began. The inlet carbon dioxide concentrations during testing were held constant either at 3.8 or 7.6mm Hg partial pressures (0.5 or 1.0 mole percent carbon dioxide at one atmosphere).

The sorbent is capable of removing carbon dioxide from streams with carbon dioxide concentrations below 2.3mm Hg (0.3 mole percent carbon dioxide) as was demonstrated previously. The following table lists the carbon dioxide concentrations and the necessary air flowrate to comprise the input rate of 40 liters of carbon dioxide per hour.

TABLE I

CO₂ Concentration and Flowrates for
40 liters CO₂ per hour (STP) Input Test Rate

CO ₂ conc. (mmHg)	Total Flowrate (liters per min)
2.3	221
3.8	133
7.6	67

The concentration of water vapor in the inlet test streams was approximately 19.8mm Hg partial pressure (2.6 mole percent at one atmosphere) except for a few tests with 10.5mm Hg as is discussed later.

6.1 CARBON DIOXIDE REMOVAL PERFORMANCE

Typical absorber carbon dioxide removal is shown in Figure 4 for a nominal 1.3 hour capacity unit. The carbon dioxide input rate in this case is 1.0 gram per minute,

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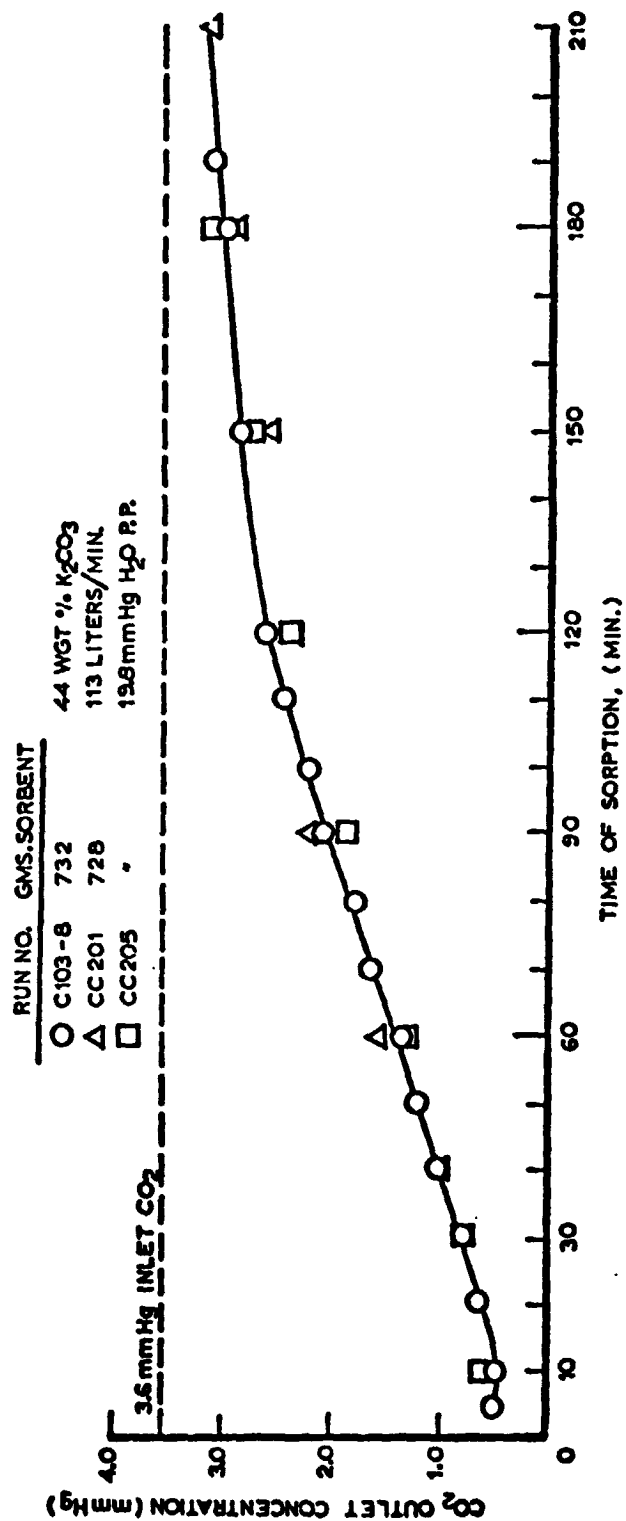


FIGURE 4 TYPICAL OUTLET CO₂ CONCENTRATIONS FROM ABSORBER TEST UNITS

approximately 33 liters of carbon dioxide per hour. The outlet concentration of carbon dioxide was maintained below 1.0mm Hg for 40 minutes with the lowest outlet concentration about 0.4mmHg.

The average grams of carbon dioxide removed per minute over 80 minutes (1.3hours) is 0.69. This corresponds to about 56.5 percent conversion of the potassium carbonate. Operation to 3.25 hours converted 75 percent of the potassium carbonate in the sorbent. This performance was reproducible (up to 15 cycles in these test units). A larger unit went on life tests up to 90 cycles and is discussed later.

However, due to the exothermic reaction and adiabatic nature of the absorber unit, the sorbent temperature increased from its initial room temperature during the period of carbon dioxide removal. The typical temperature rise behavior is shown in Figure 5. The temperature rose from 25 to 64°C peak in 12 minutes.

The temperature of the sorbent affects the amount of carbon dioxide removed and the outlet concentration. Obviously, the thermally regenerable potassium carbonate-bicarbonate reaction is dependent upon temperature, as the reaction is reversed and sorbent is completely regenerated at a temperature of 150°C.

An examination of the effect of temperature was made with single, flat sorbent sheets placed in the apparatus used in the previous study. That apparatus was a linear flow, isothermal, rectangular channel reactor. The sorbent forms a part of one wall of the channel with a gas flow channel 0.8mm high above the sorbent sheet. The dimensions of the sorbent sheet were 7.6cm x 30.5cm x 0.144cm.

Isothermal sorption runs were made with 2.3mm Hg carbon dioxide partial pressure and 19.8mm Hg water vapor partial pressure in air flowing at 1.0 liter per minute. The gas and sorbent were kept at constant temperature throughout the run. Runs were made at 0, 21, and 60°C for carbon dioxide removal.

Table II illustrates the differences in sorbent performance in terms of carbon dioxide removed at the three temperatures. The lower outlet concentration numbers in the table correspond to larger amounts of carbon dioxide removed from the air stream. For the sorbent at 60°C, 0.05gm of carbon dioxide is removed. This is 42% of the carbon dioxide removed by the same sorbent at 21°C. The isothermal performance confirms the reduced carbon dioxide pickup observed in the absorber units. It is estimated that in the absorber tests the rise in temperature of the sorbent reduces the carbon dioxide removal by about 50 percent compared to sorption at a constant 21°C. It is evident that by reducing the overall sorbent

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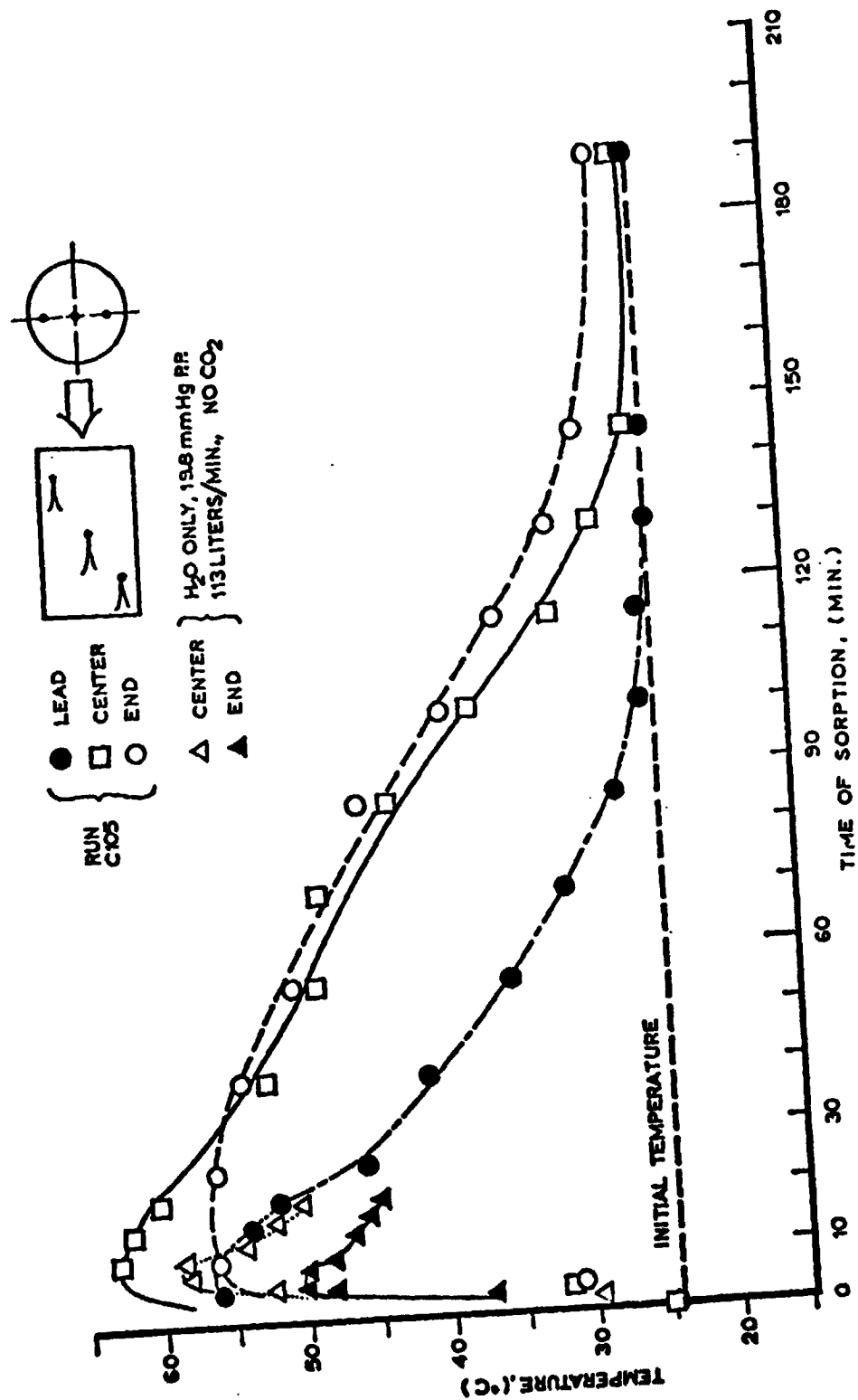


FIGURE 5 TEMPERATURE RISE AND DISTRIBUTION DURING REMOVAL OF CO₂

temperature the carbon dioxide removal would be increased. A run with 0°C sorbent temperature showed much less carbon dioxide removal indicating a lower temperature bound exists. Maximum sorption probably occurs in the 15-25°C range.

TABLE II

SINGLE SORBENT SHEET TEST IN ISOTHERMAL CHANNEL APPARATUS

Inlet Conc. 2.28mm Hg CO₂, 19.8mmHg H₂O Inlet Flow 1.0 liter/min.

Sorption Temp. (°C)	Time of Run (min)	Outlet CO ₂ Conc. (mmHg)	Gms. CO ₂ Removed after 30 mins. (grams)
0	10	2.05	0.012
	20	1.98	
	30	2.10	
21	10	0.60	0.12
	20	0.38	
	30	0.46	
60	10	1.37	0.05
	20	1.60	
	30	1.90	

The reduction of the sorbent temperature during sorption would benefit carbon dioxide capacity and achieve lower outlet concentrations. In terms of system simplicity an uncooled unit would be desired. Hence no attempts were made in this study to actively cool the absorber units during sorption. Testing was continued with the uncooled test units to explore the limits of nearly adiabatic operation. The absorber is not truly adiabatic since some heat is lost to the room and some is carried away by the exit stream.

The only controllable test parameter found that could alter the sorbent temperature was the concentration of water vapor in the inlet stream. The peak temperature diminished by 9°C by reducing the inlet water concentration from five to three times the stoichiometric required amount (19.8mm Hg is about five times stoichiometric).

Another effect of reduced inlet water content is that the amount of carbon dioxide removed in the initial period of sorption, to about 10 minutes, is slightly reduced. This is not a significant reduction compared with sorption periods of hours as long as two to three times stoichiometric water is available. Normal suit vent gases contain 15-27°C dew point moisture which is about five times stoichiometric. It is not practical to reduce the incoming water content below this amount to reduce the sorbent temperature rise.

The initial period effect is seen in Figure 6. The water inlet concentration was maintained at 10.5mm Hg partial pressure. The peak temperature reached was about 55°C, compared with 64°C, with 19.8mm Hg. Also, the different times the peak was reached are shown by square dots at the various absorber flowrates.

Figure 6 basically presents the effect of carbon dioxide input load upon the outlet carbon dioxide concentration for a given absorber unit. As the input load decreases by reducing the total flowrate, the outlet carbon dioxide concentration decreases. This illustrates the space velocity—stream conversion characteristic of the absorber unit as a chemical reactor.

In actual life support systems, the total atmosphere circulation rate may not be variable. However only portions of the total circulating air may be diverted to the absorber units to obtain performance benefits.

Some water vapor in excess over the stoichiometric requirement for carbon dioxide reaction will be removed by the sorbent. The water removed by the carbonate reaction and that removed by the formation of the carbonate hydrate amounts to 0.12 gram water per gram of total sorbent. (1)

This feature of the potassium carbonate system lessens the dehumidifier load in the life support system. By comparison the non-regenerable lithium hydroxide canisters added water to the life support system as it removed carbon dioxide.

The analysis of methods of reducing the absorber unit temperature points to the use of some direct cooling method. The heat load imposed upon the life support system would be in the same range as that imposed by lithium hydroxide system, about 80kcal per hour.

Investigation of the following factors to aid heat transfer to a cooling system would be the next step in achieving higher performance. Some of these factors are optimizing the diameter to length ratio, the addition of a center axial cooling hole or holes, splitting one unit into two, and increasing radial heat conduction. These items were not investigated in this program, only design scale—up information and baseline absorber performance were obtained.

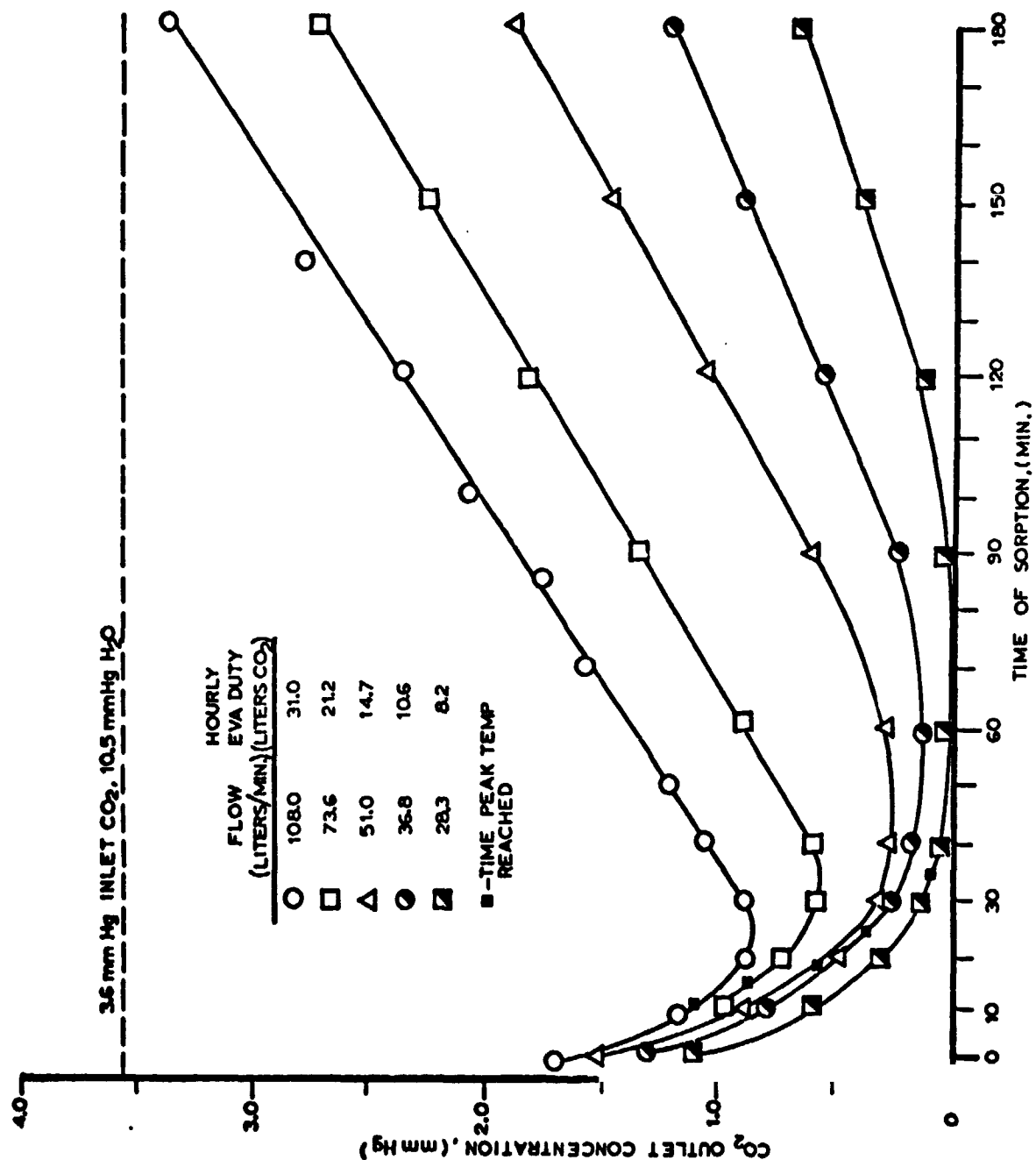


FIGURE 6 RELATION OF OUTLET CONCENTRATION WITH INPUT LOAD

6.2 REGENERATION

Two possible methods of regeneration of the potassium bicarbonate product to the potassium carbonate reactant were investigated. One method of regeneration is by simply heating the sorbent to 150°C. The other is by applying a vacuum to decompose the bicarbonate product.

Thermal regeneration works sufficiently well and conveniently. Carbon dioxide and water are given off rapidly. The rates depend on the heating rates and sensible heat build-up in the absorber material. No air flow is required other than for purging the system. The absorber test units could be regenerated in the same time or less than the sorption time.

Vacuum regeneration of the units on the other hand, was not successful. Vacuum was applied to the absorber unit for periods of hours, then the unit was thermally regenerated to drive off the remaining carbon dioxide. Measurements indicated that only about 25 percent of the carbon dioxide stored in the unit was removed by vacuum regeneration. The sorbent temperature dropped to about 10°C from 25°C indicating that heat energy was necessary for the regeneration to progress.

The hybrid regeneration system of heat plus vacuum was not investigated. The loss of the life support system gases stored in the void volume of the absorber unit could be very high with vacuum regeneration; about 50% of the absorber volume with each regeneration.

6.3 LIFE TESTING

A nominal 2.6 hour capacity absorber unit was placed on life testing to determine the effects of repeated carbon dioxide and water removal followed by thermal regeneration. The apparatus described earlier was used. The carbon dioxide inlet concentration was held at 7.6mm Hg; water at 19.8mm Hg partial pressure. The flowrate was 59.5 liters per minute STP. The temperature rise for this unit was similar to that shown in Figure 5.

A typical test cycle consisted of carbon dioxide sorption for three hours, followed by thermal regeneration by heating to 150°C. Heating was continued until no carbon dioxide was measured exiting the absorber, typically lasting for four hours. The absorber then cooled to room temperature overnight.

The sorbent performance was measured by the carbon dioxide removed upon each cycle. The carbon dioxide removed is expressed as the fraction of potassium carbonate converted at various times of sorption as shown in Figure 7. The conversion levels are reduced because of the rise in absorber temperature.

The average conversion of potassium carbonate declined about 25 percent after 60 cycles. After about 55-60 cycles, the rate of decline diminished and the conversion appears rather constant for the remaining 30 cycles up to the 90th cycle. Due to time available in the program, the testing was stopped after 90 cycles. The system goal in terms of repeated use is to have a unit capable of 100 cycles.

The cycle-to-cycle variation in conversion is probably due to ambient temperature variations and measurement errors.

A visual inspection of the sorbent cores after the 90th cycle showed no gross defects in the sorbent core. The reduction in capacity was determined in the previous program to be related to the loss of small pores in the sorbent. The composition and structure of the sorbent is promising very good cycle life.

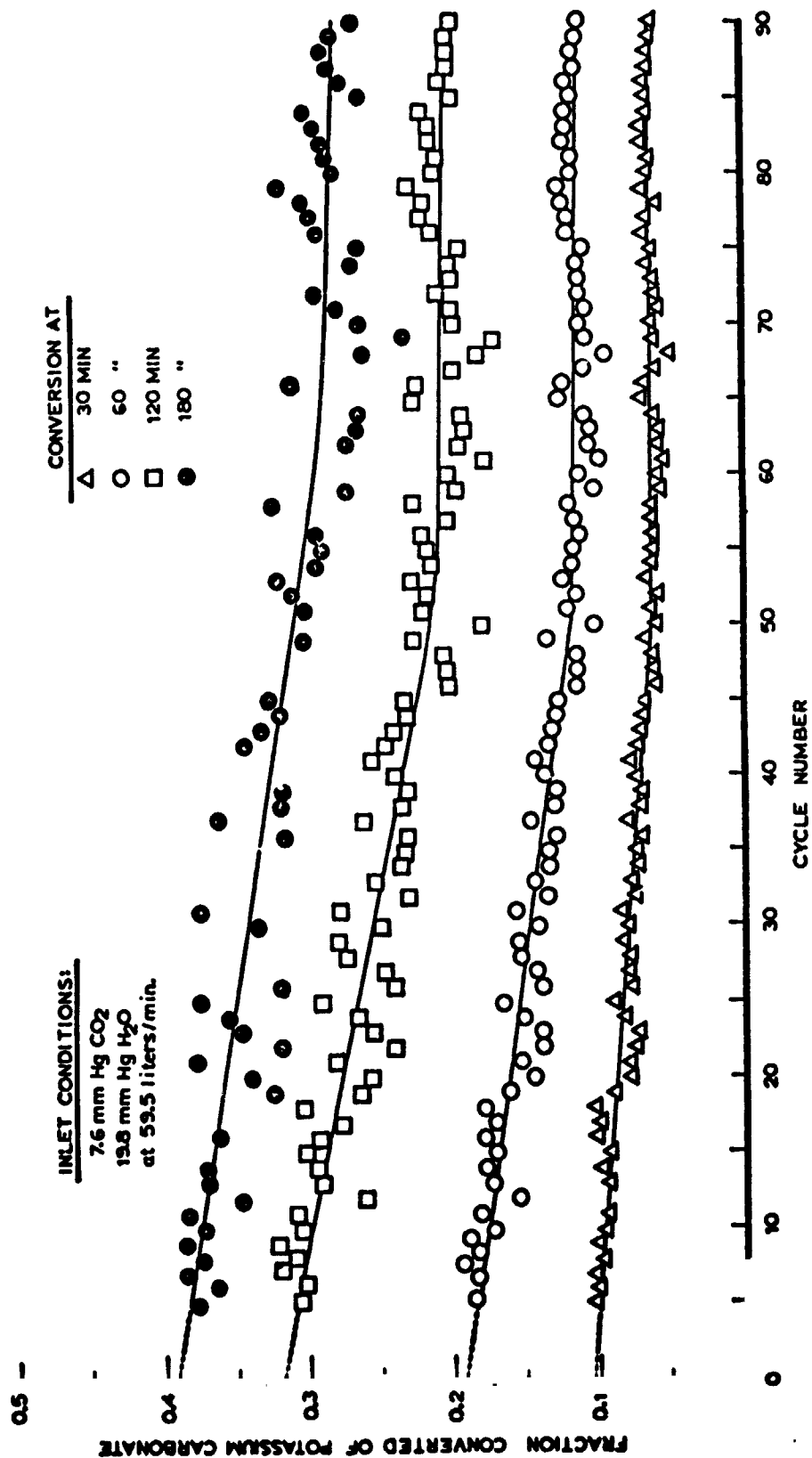


FIGURE 7 CYCLE LIFE OF AN ABSORBER TEST UNIT

7. ABSORBER DESIGN

The size and weight of an eight hour absorber unit was estimated from the performance of the absorber test units. The EVA carbon dioxide load was taken as 60 liters of carbon dioxide (STP) per hour from an average metabolic heat rate of 302 kcal per hour.

An uncooled absorber (ie, nearly adiabatic operation) and a cooled absorber unit were considered. For eight hours, the uncooled absorber requires 14.2 kg of 44% potassium carbonate sorbent material and 13.4 kg of aluminum separator. The separator weight in this design is not minimized however. The volume occupied by the sorbent material is 53.8 liters.

The conversion of potassium carbonate in this uncooled absorber is 43.5% which was the value observed during testing and is low because of the rise in sorbent temperature.

Cooling the absorber would improve its capacity, or compared to the uncooled unit its size and weight would decrease for the corresponding eight hour EVA.

If the absorber were cooled to 25°C with 90% conversion of the potassium carbonate as was indicated in the previous isothermal study, (1) then the eight hour duty sorbent would weigh 6.83kg. The separator would weigh 6.55kg and the volume occupied would be 26 liters.

However, a cooled absorber unit in the life support system carries an additional weight burden. The heat removal requirement is related, for example, to a certain amount of expandable liquid water necessary for use in an evaporative heat removal unit. Therefore, assuming a heat load of 80 kcal per hour for eight hours, 1.1kg of liquid water would be required. This must be considered in conjunction with the cooled absorber weight.

The cooled absorber unit with its added cooling load weight is lower in size and weight than the equivalent uncooled unit. Hence, cooling is advantageous in terms of lowest system size.

8. CONCLUSIONS AND RECOMMENDATIONS

A carbon dioxide absorber unit based on a new regenerable gas-solid reaction system was designed, built, and tested. The system utilized solid potassium carbonate and has features that are advantageous in the overall life support system. It can remove carbon dioxide present in the vent gases in low concentrations to absorber outlet levels below 1.0mm Hg partial pressure. It also removes water from the life support system, reducing the dehumidifier load. The regeneration of the absorber is simply accomplished by heating to 150°C, another energy saving feature.

The carbon dioxide sorbent material, formed into thin, porous sheets, was rolled between a separator screen to form a cylindrical core. The vent gases pass axially through the absorber unit.

Baseline absorber performance was established and the effects of temperature, flowrate, and inlet stream concentrations were investigated. The most important factor that adversely affected the performance of the absorber units was the temperature rise of the absorber during carbon dioxide removal.

The test units were operated partially adiabatically, with no direct cooling employed. The heat of the reaction increased the sorbent temperature from the initial 25°C to a peak of 64°C. From tests of small pieces of sorbent material held at constant temperatures of 21°C and 60°C, results show that the capacity would increase approximately 50% if the sorbent temperature were near 25°C. So with direct cooling, the absorber performance would be improved.

An absorber test unit was cycled through 90 cycles of sorption followed by thermal regeneration. The capacity reduced approximately 25% from the initial values and leveled off at that value. Regeneration by means of applied vacuum with no heating did not regenerate the sorbent satisfactorily.

To achieve design capacity with much lower outlet carbon dioxide concentrations and lowest system weight it is recommended that the absorber units be actively cooled. Also, various factors controlling heat transfer to the cooling system should be investigated to maximize its effectiveness. Among the cooling techniques to be investigated are the optimization of the D/L ratio, increasing the effective radial heat conduction, and utilizing axial cooling tubes and jackets for heat removal.

From thermodynamic values of the potassium carbonate reaction, the heat load imposed on the life support system is estimated to be the same as that imposed by the non-regenerable lithium hydroxide system used in previous missions about 80 kcal per hour. With cooling, the absorber capacity and outlet carbon dioxide concentrations will be greatly improved, providing an effective, regenerable system for carbon dioxide control.

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